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IS 1448-115 (1984): Methods of test for petroleum and its products, Part 115: Determination of salt content in crude petroleum and its products [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 115] - 1984

(Reaffirmed 2002)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 115]

DETERMINATION OF SALT CONTENT IN CRUDE PETROLEUM AND ITS PRODUCTS

(First Reprint DECEMBER 1998)

UDC 665.6/.7 : 665.61 : 543.546.331.31

Adapted with permission from the Institute of Petroleum Standard No. IP 77/72 (Reapproved 1979)

1. SCOPE

1.1 This method is intended for the determination of total halide concentration of 0·002 to 0·02 percent by mass, in crude petroleum, topped crude, residual cracking stock, and fuel oil. It may also be applied to the estimation of sea water contamination of used turbine oil and of marine diesel fuel.

1.2 The method is applicable to other products when the precision required is not greater than the limits indicated in **8**.

2. OUTLINE OF THE METHOD

2.1 The sample is extracted with water in the presence of a solvent and a demulsifying agent in a TEL extraction apparatus, IS : 1448 [P : 117]-1984* the halides in the extract are determined volumetrically after the removal of sulphide. The result is expressed as sodium chloride.

3. APPARATUS

3.1 Extraction Apparatus — Same as given under **4.1** of IS : 1448 [P : 117]-1984*.

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*Determination of lead content in gasoline by volumetric chromate method.

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Gr 1 *May* 1984

4. REAGENTS

4.0 All reagents shall be free from halide.

4.1 Acetone — (*See* IS : 170-1976*.)

4.2 Alcohol — absolute ethanol (*see* IS : 321-1964†) or absolute ethanol denatured by the addition of not more than 5 percent methanol.

4.3 Iso-amyl Alcohol — (*See* IS : 360-1964‡.)

4.4 Toluene – (*See* IS : 1839-1961§.)

4.5 Lead Acetate Papers

4.6 Nitric Acid 5 N — (*See* IS : 264-1976||.) Dilute 1 volume of concentrated nitric acid with 2 volumes of distilled water, and boil until it is colourless. Cool and blow the solution with air to remove oxides of nitrogen.

4.7 Potassium Thiocyanate Solution — 0·05 N, accurately standardized.

4.8 Silver Nitrate Solution — 0·05 N, accurately standardized.

4.9 Ferric Alum Indicator — Dissolve 350 g of ferric ammonium sulphate in 1 litre of distilled water and add 200 ml of 5 N nitric acid.

5. PREPARATION OF SAMPLE

5.1 The difficulties in obtaining representative samples for this determination are considerable and hence the importance of correct sampling cannot be too strongly emphasized. Observe the precaution indicated in the appropriate sections of IS : 1447-1966¶ and do not use the water displacement method for sampling.

6. PROCEDURE

6.1 Weigh 80 ± 0·5 g of the sample into a 250-ml beaker and heat it to 60°C ± 5°C. Heat 40 ml of toluene to the same temperature and add it slowly to the sample with constant stirring until solution is complete. Transfer this solution quantitatively to the flask of the extraction apparatus through the thistle tube, and wash it in with two 15-ml portions of toluene, each at about 60°C.

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*Specification for acetone (*first revision*).

†Specification for absolute alcohol (*revised*).

‡Specification for amyl alcohol (*revised*).

§Specification for toluene, reagent grade.

||Specification for nitric acid (*first revision*).

¶Methods of sampling of petroleum and its products.

CAUTION — Toxic vapour; adequate ventilation should be provided.

6.1.1 Without allowing the mixture in the flask to cool, add 25 ml of hot alcohol and 15 ml of hot acetone.

6.2 Boil the mixture vigorously for 2 minutes, remove the source of heat and when boiling has ceased add 125 ml of distilled water. Boil the mixture for a further 15 minutes.

6.3 Allow the phases to cool and separate. Draw off the lower layer and filter it, if necessary, through a Whatman No. 41 or equivalent filter-paper. (Because some alcohol and acetone remain dissolved in the toluene phase, the volume of extract available is taken as 160 ml).

6.4 Measure into a beaker 100 ml of the extract and add 5 ml of the nitric acid. Cover the beaker with a clock-glass and bring the contents to the boil. Test the vapours periodically for hydrogen sulphide with lead-acetate paper and continue to boil for a further 5 minutes after a negative result is obtained. Cool and then rinse the contents of the beaker into a titration flask with distilled water. Add 10 ml of *iso*-amyl alcohol and 3 ml of ferric alum indicator.

6.5 From a burette add 0·5 ml of the thiocyanate solution. Titrate the mixture with silver nitrate solution to a colourless end point, agitating the contents of the flask continuously, and add an excess, of 5 ml of the silver nitrate. Stopper the flask and shake it vigorously for 15 seconds to coagulate the precipitate (*see* note).

NOTE — Care shall be exercised in opening the flask. During shaking, pressure may develop and this may cause small amounts of the acid solution to be sprayed from the mouth of the flask as the stopper is removed.

6.6 Titrate slowly with the thiocyanate solution until the end point is approached, as indicated by slower fading of the red colouration. Shake the flask again and continue the titration drop-wise until a red colouration persists after vigorous shaking.

6.7 Make a blank titration by repeating the procedure, replacing the 80 g of sample with 95 ml of toluene.

7. CALCULATION AND REPORT

7.1 Calculate the halide content of the sample either as percentage sodium chloride by mass, or as milligrams per litre:

a) NaCl by mass, percent $= 9{\cdot}35\,[\,(V_1 - V_2)\,N - (V_3 - V_4)\,n\,]/M$

b) NaCl per litre, mg $= 9{\cdot}35 \times 104\,G\,[\,(V_1 - V_2)\,N - (V_3 - V_4)\,n\,]/M$

where

V_1 = volume of silver nitrate solution used to titrate the sample;

V_2 = volume of silver nitrate solution used to titrate the blank;

V_3 = volume of thiocyanate solution used to titrate the sample; including the 0·5 ml added in **6.5**;

V_4 = volume of thiocyanate solution used to titrate the blank, including the 0·5 ml added in **6.5**;

N = normality of the silver nitrate solution;

n = normality of the thiocyanate solution;

M = mass of sample in grams; and

G = relative density of the sample at 15·5°C/15·5°C.

7.2 Report the result to the nearest 0·000 2 percent or to the nearest 2 mg per litre as the Salt Content.

8. PRECISION

8.1 Results of duplicate tests shall not differ by more than the following amounts:

Repeatability	*Reproducibility*
0·002 percent or 12 mg/litre	0·004 percent or 24 mg/litre